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QUALIFICATION OF NONMETALLIC MATERIALS FOR THE VIKING MARS LANDER

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ABSTRACT

The Viking Mars mission has several unique environmental aspects that impose requirements on materials greatly surpassing those of previous space missions. Foremost among these requirements are: (1) the ability of all materials in the spacecraft to withstand exposures to 125°C for up to 100 hours without any deleterious effects; (2) a minimal or predictable change in material properties after 10 to 14 months exposure to the thermal vacuum environment of interplanetary space; (3) noninterference by materials of life detection and organic analysis experiments to be conducted on the surface of Mars. These requirements formed the basis for an elaborate materials qualification program. The elements of this program, including philosophy, test methods and facilities, are described.

INTRODUCTION

An extensive material qualification test program has been conducted to ensure the proper selection, processing, and application of nonmetallic materials in the design and construction of the Viking Mars Lander. As a part of the program, thousands of tests were conducted to derive basic thermochemical data, degassing data, and critical physical property data of nonmetallic materials ranging from marking inks to heat shield materials.

The vast amount of data obtained from this program will have significant value for both future aerospace programs and certain non-aerospace commercial activities.

Viking nonmetallic materials must be capable of withstanding the environmental conditions the probe will encounter before during and after its journey from earth to Mars. This includes sterilization temperatures of 125°C for periods of 100 hours or more, thermal vacuum exposures for periods of 14 months, and the ability to withstand the Martian environment without degradation or decomposition sufficient to interfere with the science package aboard the Viking Lander. Such areas as the soil sampling system, the organic analysis experiment, and the detection system have strict

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requirements regarding the allowable organic contamination permissible as background in these systems.

A very significant aspect of the program described in this paper is in the discipline and controls employed. No other major hardware material qualification program to date has dictated the rigid control on facilities, methods, and materials as has occurred in the Viking Program. One reason for this strict discipline is that the qualification testing on any one material is extensive, and in comparing data from different lots and batches with the material originally qualified, no question should arise as to the methods or facilities employed. A second reason for the discipline in this program is the shear magnitude of the effort. If careful recordings of each test were not maintained in an organized, disciplined fashion, the total volume of data would make it virtually impossible to reconstruct the test history associated with any single material. Finally, for any material tested, the material processing history and traceability up to the point of testing is of great significance since the chemical or physical behavior of polymers and other nonmetallics can be very dependent on prior treatment.

A significant fraction of the Viking Materials Program involves the selection of nonmetallic materials, since these materials as a class are considered most vulnerable to the Viking mission environment. This selection process for nonmetallics involves both chemical and physical property determinations. Considerable effort has been spent in the screening of candidate materials, the qualification of successfully screened materials, and the tracking or fingerprinting of qualified nonmetallics through hardware production.

MATERIAL SELECTION AND CONTROL

A candidate materials list was generated for the qualification program by using the following sources: 1) prior space vehicle history; 2) prior laboratory screening tests in other laboratories; and 3) preliminary in-house laboratory screening tests. Materials having little or no prior history for which a definite need was established on the program were screened in-house using vacuum thermogravimetric analysis combined with residual gas analysis. These specific tests, being relatively inexpensive and of short duration, give a good deal of information relating to material's thermal stability, degassing properties, cure effectiveness, resin content, etc. A further advantage of this technique is the small sample (12 to 20 mg) required for testing.

All candidate materials were assigned a specific identification number. Each material lot/batch was assigned a different number, even though the material may have been obtained from the same source and was supposed to be the same material type. Using

this method, it was possible to trace all the information regarding material, process, preparation, testing and qualification or rejection of any given lot or batch. Once the material had been subjected to all the necessary testing, a complete file of the test data was presented to a material review committee whose function it was to pass judgment on unlimited use, restricted use, or rejection for use on the Viking Lander.

A formal test report was generated for each material. This report contained a summary of the material history, test data compilation for both chemical and physical properties, and the conclusions reached by the review board. As a result of this action, material and process specifications were implemented to ensure the integrity of the materials used throughout the Viking Lander.

PROGRAM DESCRIPTION

Chemical Characterization

The chemical characterization phase of the qualification program consisted of the following tests: thermal degradation, mass spectral analysis, condensible outgassing, isothermal weight loss, and gas chromatograph-mass spectrometer analysis.

Thermal Degradation--Using thermogravimetric analysis (TGA), degradation testing from ambient temperature to 500°C or above was performed in a vacuum and, in some cases, nitrogen. The vacuum environment is representative of service, whereas the nitrogen simulates the sterilization environment.

Mass Spectral Analysis--To characterize the outgassing and degradation species during the TGA test, a residual gas analyzer was attached to the TGA apparatus to provide simultaneous mass spectral data during the vacuum TGA test.

Condensible Outgassing--The rate of deposition of condensibles from candidate materials was determined using a quartz crystal microbalance operating at -125°C in vacuum, the minimum temperature anticipated at the Martin surface.

Isothermal Weight Loss--To assess materials behavior during the sterilization cycle, isothermal weight loss tests were performed at 135°C for 100 hours in a nitrogen atmosphere.

Gas Chromatograph-Mass Spectrometer Analysis--For those materials used in the spacecraft in critical locations or in significant quantities, the material will be pyrolyzed and a gas chromatograph-mass spectrometer reference spectra generated. Testing of this type began in 1973.

A listing of the types of materials tested in this portion of the program is given in Table I. This table includes materials which did not get beyond the screening stage, but does not include "fingerprint" testing of different lot/batches of the same material.

Table I - Quantity of Nonmetallic Materials Chemically Tested

Generic	Quantity	Application	Quantity
Acrylics	2	Ablator	13
Butyl Rubber	6	Adhesive	127
Diallyl Phthalates	18	Coating	73
Epoxies	307	Elastomer	64
Ethylene Propylene Rubber	5	Electrical Insulation	110
Fluorocarbons	60	Encapsulant	65
Miscellaneous (principally inorganic nonmetallics)	92	Fabric	57
Phenolics	13	Film	25
Polyamides	20	Finish	12
Polycarbonates	1	Lubricant	24
Polychloro-P-Xylene	2	Marking	26
Polyester	43	Miscellaneous	20
Polyethylene	5	Molding Compound	9
Polyimide	45	Sealant	36
Polyolefin	2	Shock Insulation	10
Polypropylene	7	Structural Plastic	45
Polystyrene	5	Tape	18
Polyvinyl Chloride	1	Thermal Insulation	48
Polyvinyl Fluoride	6	Tubing	13
Silicone	123	Wire	18
Unknown	11		
Urethane	39		
Total	813	Total	813

Thermal Degradation TGA-RGA in Vacuum

A thermogravimetric analysis (TGA) combined with residual gas analysis (RGA) test is a short duration test which gives much information relating to the material's thermal stability, degassing properties, cure effectiveness, resin content, and the activation energy for thermal degradation. A further advantage of this technique is that only a very small sample (6 to 10 mg) is required for testing. Following sample conditioning, the specimen was heated in vacuum at a programmed rate of 10°C/minute to a maximum of 500°C or to the point at which decomposition is complete, whichever occurred first. Sample weight was continuously recorded throughout the heating cycle. Mass spectral data of evolved gases in the mass range m/e 2 to 250 was obtained at 10°C to 15°C intervals. RGA data from the screening test can be reduced in graphical form showing the change in relative peak heights

of significant m/e peaks (not present in background) versus sample temperature.

TGA-DTA in Nitrogen

In some cases, TGA-DTA tests were performed in a continuous nitrogen gas flow and other gaseous environments. In these tests Differential Thermal Analysis (DTA) was substituted for the RGA. This nitrogen testing was performed to determine whether the thermochemical behavior of the polymer is influenced by the presence of nitrogen (a common sealing gas). Samples were prepared, cleaned, and conditioned in the same manner as in the vacuum TGA screening test. TGA data can be reduced to a percentage weight loss as a function of temperature. DTA data can be reduced to give the temperature at which significant endothermic or exothermic reaction occurs.

Isothermal Weight Loss Prediction from TGA Data

Thermal degradation under vacuum, as measured by isothermal weight loss of polymers, has been studied for a variety of polymers by H. A. Papazian and his coworkers[1]. Small samples of nonmetallic materials have been used in the TGA tests. Isothermal studies, at much lower temperatures, have been made using larger samples (6.0 to 10.0 grams). Comparison of the two techniques has shown that the TGA result can accurately predict the isothermal kinetics.

Using the Arrhenius relation,

$$k = A\, e^{-\Delta E/RT} \tag{1}$$

and, accordingly,

$$\ell n\, k = \ell n\, A - \frac{\Delta E}{RT} \tag{2}$$

where k is the rate constant, A is known as the pre-exponential factor, and ΔE is the activation energy.

In the study of thermal stability of nonmetallic materials, the degradation reaction was found, in most cases, to be a first order reaction, and the degradation rate is directly proportional to concentration of the reacting substance,

$$-\frac{dc}{dt} = k\, c \tag{3}$$

where c is concentration of the reaction species, t is time. Let "a" be the initial weight of nonmetallics, and "x" the weight after time "t". The remaining weight is (a - x), and

$$-\frac{d(a - x)}{dt} = k(a - x) \tag{4}$$

Therefore,

$$\frac{dx}{dt} = k(a - x) \tag{5}$$

From equation (1), the activation energy in Kcal/mole is

$$E = 4.574 \times 10^{-3} \frac{d(\log k)}{d\left(\frac{1}{T}\right)} \tag{6}$$

Integration of equation (5) yields

$$\frac{a - x}{a} = e^{-kt} \tag{7}$$

where a is the initial weight and a - x/a is the fraction remaining. Therefore, if k is known for a given temperature, one can find the fraction of the polymer remaining for a predetermined time, t, or if the weight loss is desired,

$$(1 - e^{-kt}) \times 100 = \% \text{ weight loss} \tag{8}$$

Material Fingerprinting

To assure identicality of materials used in the hardware phase of Viking with initial lot/batches of qualified materials, all later buys of most nonmetallics were subjected to a TGA-RGA "fingerprint" test as part of the receiving inspection function. In this manner, all new lots of material could be compared with the original qualification lot to ascertain that, chemically, the material and processing of that material were identical to the original qualification lot. This technique, put to test on over 800 materials to date, has proven to be an extremely sensitive tool in detecting small process variations.

Condensible Outgassing Tests

For measurement of outgassing, a standard method developed for use in the Viking Program is used. In this test, a quartz crystal microbalance is used to measure the rates of condensible degassing issuing from the sample. With the sample heated in an effusion cell, a cooled quartz crystal is located in close proximity to the orifice of the cell. As the degassed species issue from the cell and impinge on the surface of the quartz crystal, the condensible portion of the sample degassing

collects on the quartz, while the noncondensibles are pumped from the system. The sensitivity of this method permits condensible degassing rates as low as 1×10^{-5} percent/day to be accurately determined.

Nitrogen Weight Loss

The nitrogen isothermal weight loss tests were performed in accordance with a standard method developed for use to detect the material behavior during the heat sterilization cycle. The weight of the sample was measured before and after the 100 hours at 135°C with a 3 cc/minute nitrogen gas purge. The nitrogen purge outlet was cold trapped at -196°C during the test and then warmed up to room temperature while taking a residual gas analysis scan. For most materials, the condensibles were only air constituents.

GCMS Analysis

Those materials selected by the Viking Science Team to be used in the spacecraft in critical locations such as soil sampler assembly or in significant quantities will be pyrolyzed and a GCMS reference spectra determined. Tests of this type are to begin in the latter part of 1973.

Typical Qualification Test Summary (Chemical)

An example of a data summary of the thermochemical qualification testing of a typical nonmetallic, including TGA and DTA data (Figure 1) and mass spectral data (Table II) is as follows:

Material, EA934--EA 934 adhesive is a two part epoxy used for general purposes. This material is governed by MMA Viking Document TP 3720150 for all the control, specimen preparation and testing.

Vacuum, TGA--Onset of major degradation, 220°C - 235°C (493°K - 508°K). Weight loss at 135°C (408°K) ranges from 0.3 to 0.9% and consisted almost entirely of water loss. This loss occurred over the temperature range of 50°C - 135°C (323°K - 408°K). The highest water loss occurred in the specimen preconditioned at room temperature and 45% RH, while the lower water contents were observed in specimens preconditioned at 125°C (398°K) in nitrogen for 100 hours (3.6×10^5 sec).

Nitrogen, TGA--Onset of major degradation, 240°C - 265°C (514°K - 538°K). Weight loss at 135°C (408°K) was 0.45%. This loss occurred over the temperature range of 55°C - 140°C (328°K - 413°K). The specimen was preconditioned at room temperature and 45% RH for minimum of 24 hours prior to test.

Nitrogen Weight Loss--The weight loss in nitrogen after 100 hours

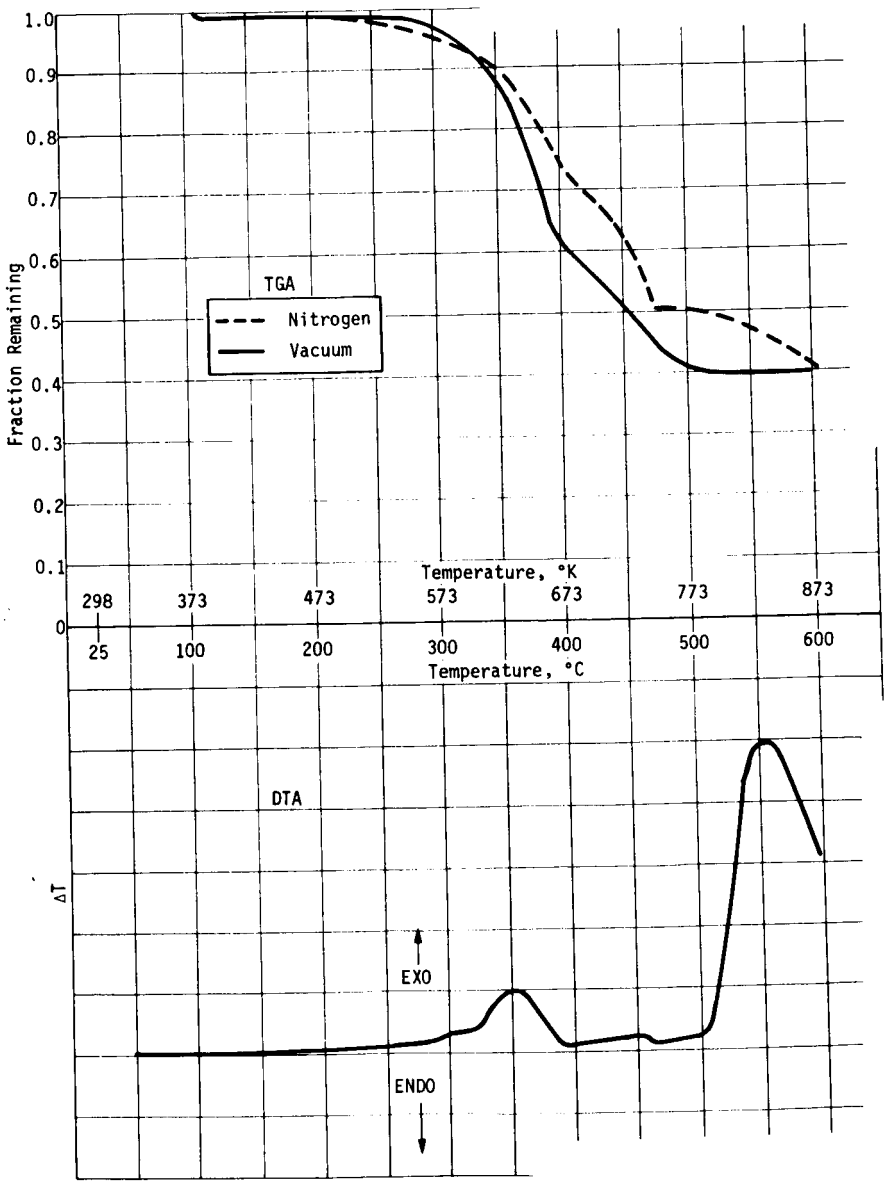


Figure 1 - TGA-DTA Curves for Epoxy Adhesive, EA 934

Table II

EA 934 Mass Number and Relative Peak Intensity

m/e	Temperature, °C (298°K) 25	(573°K) 300	(623°K) 350	(673°K) 400	(723°K) 450	(773°K) 500	(823°K) 550
14	560	796	3 022	1 230	1 152	1 386	1 248
15	129	827	8 306	2 397	2 429	3 039	3 052
16	1 851	3 741	14 236	4 393	3 943	4 498	5 071
17	12 954	18 467	41 298	15 094	11 576	10 344	10 217
18	48 262	65 048	100 364	49 595	39 877	36 059	36 148
19	42	118	176	127	42	55	41
20	89	127	276	143	109	100	117
21							
22							
23							
24			422	100	108	117	41
25		95	1 952	663	580	564	191
26	44	993	13 180	4 443	4 366	4 043	1 479
27	76	1 044	16 218	6 197	7 465	6 903	2 053
28	10 230	13 538	36 439	18 124	17 149	16 609	13 172
29	106	776	7 677	2 938	5 325	4 851	821
30		154	9 887	1 916	873	736	349
31		127	2 837	399	109	64	105
32	2 530	2 546	2 844	2 420	2 258	2 319	2 188
33			51				
34							
35							
36			149	60	44	42	
37		46	1 946	509	290	273	72
38		106	4 043	1 464	795	606	152
39		420	13 088	4 760	5 291	4 850	919
40	996	1 594	8 671	3 407	2 761	2 600	1 557
41		282	6 449	3 182	7 262	7 060	724
42		372	9 469	2 182	3 379	3 037	370
43		571	6 056	2 672	5 871	4 318	326
44	155	2 544	9 614	1 810	1 219	743	286
45		101	1 572	192	58		45
46			118	41			
47			289	88			
48			72				
49			539	176	100	95	
50		78	3 204	1 550	792	522	142
51		95	3 616	1 998	1 091	768	188
52		110	2 867	1 049	469	329	117
53		84	3 193	1 613	1 124	828	128
54		42	1 375	456	501	427	68
55			2 255	1 398	2 430	2 440	165
56		50	1 853	699	1 937	1 718	155
57			1 185	585	1 878	1 365	67
58		54	1 889	177	149	100	
59			340	40			
60			88	50			
61			213	122	51		
62			549	224	79	59	
63			1 256	721	284	172	103
64			652	153	66	68	
65			3 294	1 630	566	280	101
66		54	4 366	1 686	365	210	94
67		111	2 853	326	529	634	73
68			353	128	173	186	
69			170	85	362	457	
70			230	126	553	487	
71			147	68	274	237	
72			139	42	52		
73			116				
74			204	76			
75			81	61			
76			127				
77			1 230	1 291	713	344	84
78			462	230	131	128	46
79			1 027	727	388	228	65
80		40	1 151	547	212	139	
81			377	105	134	163	
82			73	50	100	85	
83			52		60	97	
84			86		98	64	
85					73	73	
86							
87							
88							
89			54	56			
90			80	59			
91			209	242	197	143	42
92			74		44	49	
93			269	63	57		
94		48	4 030	1 230	202	84	
95			257	43			
96							
97							
98							
99							
100							
101							
102							
103							
104							
105							
106							
107							
108			560	278	139		
109			315	182	54		

(3.6×10^5 sec) exposure, at 135°C (408°K) was 0.75%.

Vacuum Condensible Degassing Rate--The steady-state vacuum condensible degassing rate for this material at 125°F (323°K) is 9.13×10^{-5} percent/day.

Activation Energy of Decomposition--In vacuum:

Over the range 233°C - 300°C (506°K - 573°K) -

$A = 8.3 \times 10^{12}$

$\Delta E = 15.77$ Kcal (75.77 Kjoules)/mole

Over the range 300°C - 345°C (573°K - 628°K) -

$A = 2.1 \times 10^{12}$

$\Delta E = 56.44$ Kcal (225.35 Kjoules)/mole

In nitrogen:

$A = 5.6 \times 10^{12}$

$\Delta E = 17.27$ Kcal (72.01 Kjoules)/mole

Over the range 322°C - 370°C (596°K - 643°K) -

$A = 2.56 \times 10^{12}$

$\Delta E = 46.84$ Kcal (195.32 Kjoules)/mole

Time for 1% Weight Loss at Temperature, T

Temperature	Time (Minute)* In Vacuum	In Nitrogen
50°C (323°K)	782.2	52.5
100°C (373°K)	173.2	17.2
150°C (423°K)	54.8	7.1

*For larger sample applications, a factor of $\sim 10^2$ has to be applied to all the time in minutes.

Physical Properties Characterization

The qualification of the nonmetals used on the Viking Program with respect to their physical properties (mechanical, electrical, thermal, and optical) was a significant effort. Since there was a limited quantity of in-situ data available on the properties of materials after long term exposure in a high vacuum, it became necessary to institute an evaluation and qualification program

that would provide this necessary data after exposures to simulated space environments over extended periods of time up to 14 months. This meant that materials would have to be stored in vacuum chambers for extended periods of time and then tested in-situ. That is, the testing would be performed in a vacuum at the anticipated thermal conditions that could be expected during the flight to Mars as well as after landing on the planet.

The initial program identified a minimum of 75 different candidate materials. This amount increased during the program to over 800 materials. To evaluate these 75 materials could involve as many as 20,000 tests. However, as the program progressed, certain tests were eliminated for various reasons, such as testing for only those properties which were critical to the specific material application rather than all the physical properties of each material. In many cases, the long term testing was eliminated if the material showed little or no change over a 3 to 6 month vacuum exposure.

A sequence of testing was generated to provide a meaningful comparison of materials properties throughout the program. This sequence required the testing of materials as follows:

1. Baseline (as received);
2. Prelaunch (after receiving a heat sterilization);
3. Launch, cruise, and deorbit (after vacuum exposures of one, three, six and 14 months).

Once an acceptable program for material qualification was identified, the facilities required for this program were evaluated. The first two phases of testing (baseline and after-heat sterilization) required little more than conventional testing equipment and ovens. The third and fourth phases (vacuum soak and in-situ testing) presented severe problems. Conventional testing facilities were evaluated. Such techniques included a carrousel fixture, where a quantity of specimens could be placed in a rotatable fixture and placed in a vacuum chamber. The disadvantages of this arrangement included price and long term reliability. Each test would require special fixtures for that test and, for instance, if flexure testing was required on 20 different materials, then 20 flexure rigs and vacuum chambers would be required and the testing machine would have to be physically transferred from chamber to chamber. If all the specimens were in one chamber, then a power failure or any equipment failure would ruin the entire set of specimens. If this occurred, let's say after five months of vacuum exposure, then it would be necessary to start the test sequence (including the heat compatibility cycle) over again.

The method selected for the vacuum soak and in-situ testing was to separate the two tasks. To accomplish this, 32 vacuum canisters were fabricated. The soak systems consisted of four

400 ℓ/sec ion pumps with a seven-canister manifold on each. Each canister was separated from the plenum with two vacuum valves. The remaining four canisters were connected to individual 50 ℓ/sec ion pumps. This provided the capability of using individual canisters for those materials that either had anticipated high outgassing loads, or the outgassing products were undesirable contaminates for other materials. Figure 2 shows one of the seven canister systems. Each of the canisters was fabricated from stainless steel with a double wall to provide thermal capabilities. A schematic of these canisters is shown in Figure 3. The top portion or lid, was sealed with an O-ring compressed by a marman clamp. This sealing technique provided a full opening lid that required the tightening or loosening of only one nut to open or close the canister.

The test chamber was designed and fabricated to be used in conjunction with the soak systems. It is constructed of 300 series stainless steel and consists of two individual vacuum chambers separated by a 24 inch sliding gate valve (Figure 4). The main chamber is a nominal 5 feet in diameter and 7 feet long. The air lock chamber is 2 feet in diameter and 2 feet long.

The pumping portion of the main chamber consists of two 20-in. oil diffusion pumps with a multicoolant baffle and a valve isolating the pump from the chamber, and a 300 cfm roughing pump on the discharge side of the diffusion pumps. Fifteen cfm holding pumps are tied into the roughing manifold to enable the diffusion pumps to be exhausted while the main chamber is being roughed down. The net pumping speed for the main chamber is in excess of 10,000 ℓ/sec. The air lock pumping systems consist of a 6 inch diffusion pump with a multicoolant baffle and an isolation valve.

The 6 sq ft chamber window has three tempered glass sections, each laminated with two layers of 0.75 in. thick glass. Twenty-nine flanges on the main chamber range in size from 1.5 to 8 in. tube size. The flanges are fitted with feed-throughs for high voltage, coaxial, high current, instrumentation, liquid nitrogen and nude ion gages. A full opening door on one end provides easy access to the chamber. The air lock has four 1.5-in. tube size flanges and a sliding tray mounted on tracks within the air lock.

The unique feature of this system is the master/slave manipulation capability in the main chamber. The manipulators are similar to those used in nuclear installations and each consists of four major parts: the master arm, the slave arm, the seal tube assembly, and the tongs. Each slave arm contains over 70 bearings, lubricated with a dry film lubricant. Tong configurations can be changed remotely using a special fixture. The manipulators provide six degrees of freedom and have electric indexing in two axes for displacement of the master arm relative to the slave arm. All other motions are mechanical, with a one-to-one force ratio between the master arm and the slave arm except for

Figure 2 - Seven Canister Thermal Vacuum System

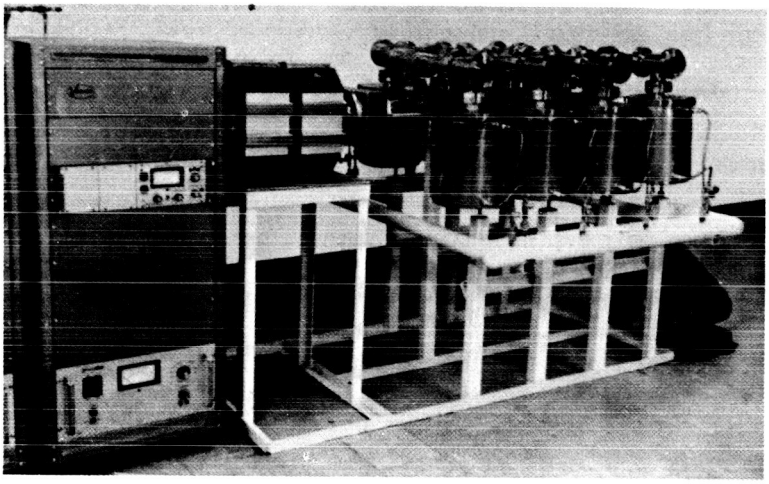


Figure 3 - Vacuum Canister

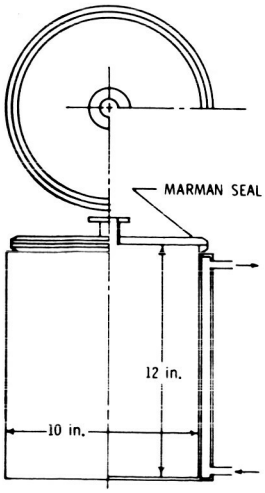
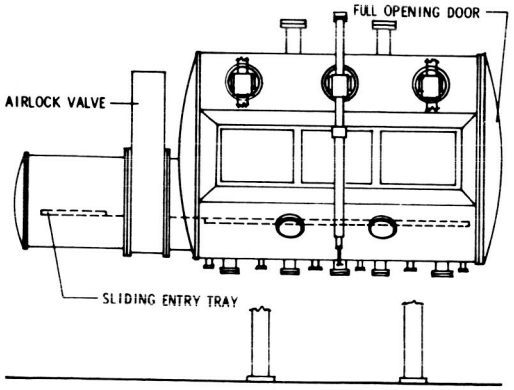
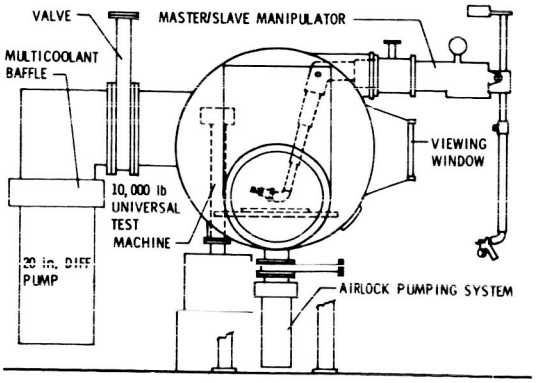


Figure 4 - Vacuum In-Situ Test Chamber





the friction of the motion rods within the seal tube assembly.

The linear motions of the master arm are converted to rotary motion at one end of the seal tube and then translated back to linear motion at the slave end of the seal tube assembly[2]. This conversion enables the use of rotary seals to maintain a vacuum. Each end of the motion rods within the seal tube contains twin Viton seals, with the cavity between each set of seals filled with low pressure oil for lubrication. The chamber routinely can be evacuated to 10^{-6} to 10^{-7} torr.

A 10,000 lb universal test machine has been coupled to the main chamber. The columns are shock isolated from the chamber with bellows, and the moving crosshead pull rod is attached to a bellows with a 14-inch stroke capability. Heating and cooling of the test specimens is provided by radiant heaters (quartz lamps) and liquid nitrogen cooled shrouds. A typical sequence for the removal of a specimen from the soak system and the in-situ testing is as follows:

- Isolate the soak canister from the manifold by closing both the vacuum valves;
- Remove the canister and mount it on the sliding tray from the air lock;
- Install the sliding tray/canister in the air lock and evacuate the air lock;
- With the main chamber under vacuum, open the 24" gate valve that separates the air lock from the main chamber;
- Reach in the air lock with the master/slave manipulator and pull the sliding tray into the main chamber;
- Loosen the nut on the marman clamp and open the canister lid;
- Remove the specimens to be tested and close the lid;
- Push the canister back in the air lock, close the gate valve, and vent the air lock;
- Remove the canister and reinstall it on the soak system;
- Evacuate the area between the ion pump and the canister and open the canister isolation and ion pump isolation valves.

The test program required the capabilities and fixtures for performing over 40 different types of tests. Table III shows the different physical properties tests that were performed on the Viking program. Approximately 85% of these tests were performed in vacuum. These tests were performed generally according to ASTM test methods; however, in certain instances, modifications were made to the methods or special test methods were generated. The

Table III - Physical Properties Measured for Viking Materials

1. Tensile - (-150 to +600°F) - D412, D638, D882, D1000
2. Lap Shear - D1002, D1000
3. Flexure - D790, FTMS 141M6221
4. Peel Tests - D1876, D1867, D1000, D903
5. Bond Tension - D952
6. Tear Tests - D624
7. Dimensional Stability - C548, D1204, D2126
8. Compression Set - D395, FTMS 601M3311
9. Scratch Tests
10. Bend Tests

   } Adhesion - FTMS 141A Method 6304.1, D1000

11. Sand and Dust Abrasion Tests - STM*
12. Dielectric Constant - D150, D1673
13. Loss Tangent - D150, D1673
14. Surface and Volume Resistivity - D257, D1000
15. Dielectric Breakdown - D149, D1000
16. α_s, E Values for Thermal Control Coatings - STM
17. Insulation Resistance - D257
18. Compression Strength - D695, C165, D1621
19. Hardness - FTMS-M3021, D785, ASTM D2240
20. Thermal Expansion - D696
21. Specific Gravity - D792*
22. Solvent Resistance - D471*
23. Thermal Cond - C177
24. Creep - D674
25. Bearing Strength and Modification - FTMS 406M1051
26. Water Absorption - FTMS 406M7031*
27. Corona Resistance - D1868
28. Moisture Resistance - D570*

* Tests not performed in vacuum.

test method for surface and volume resistivity, for instance, called for the electrodes to be secured to the specimen by an adhesive or grease. The method was modified for use in the vacuum chamber and a constant force was applied to the specimen electrode configuration. By performing the entire sequence of tests using this method, a relative change could be determined between baseline and in-situ testing if one existed. Other test method modifications were required where measurements were needed on specimens at other than ambient temperatures. Because radiant heat transfer is the primary method of heating and cooling in a vacuum, it was necessary to fully enclose the test specimen within a cooling shroud to minimize temperature gradients on certain test specimens. Head travel was used to measure elongation on those tensile specimens where strain was a critical property. Again, this technique provided a relative change if one existed. Dielectric strength tests required reduced power testing due to corona discharge problems. A 50 KV machine was used for these tests, but corona discharge occurred in vacuum between 30 and 40 KV. This problem could have been corrected if time permitted. The corona occurred over the surface of the specimens and, if new specimens and fixtures were fabricated with a much larger surface area, it is felt this problem would have been corrected. This voltage was sufficient, however, to qualify the materials for the Viking program, since breakdown did not occur through the material.

In conclusion, it is felt that the materials qualification program performed was a meaningful one, and did categorize those materials that exhibited changes after extended periods of time in a vacuum. Most materials showed little or no change after this vacuum exposure, and those that did either were rejected from the program or it was determined the changes were not detrimental to the materials specific application. Teflon impregnated fiberglass lacing cord, for instance, showed a 41% increase in tensile strength after 3 months in vacuum. CR-124 (a potting compound) showed a change in the measured resistance from 10^{13} ohms to 10^{4} ohms after heat compatibility. This change was sufficient to have the material rejected from the program.

DISCUSSION

In the chemical qualification of nonmetallic materials, the techniques employed most often from the material initial screening through qualification and in the final lot to lot fingerprinting were thermal gravimetric analysis (TGA), and combined residual gas analysis (RGA). The success of this program is due in large measure to the instruments used; namely, a Mettler Thermoanalyzer I, and Quadrupole Mass Spectrometer. The RGA data were obtained simultaneously with the TGA and read-out through an Infotronic CRS-160 Data Acquisition System.

Based on the experience of running more than 1500 tests, this method as a basic part for nonmetallic materials characterization

or qualification is highly recommended. For getting identical results from lot to lot samples, it is recommended that this tool become a part of the quality receiving/inspection process for nonmetallic materials.

In the physical properties portion of the qualification program, several conclusions can be reached. First, the testing of nonmetallic materials for obtaining relative changes after long term thermal vacuum exposures has categorized certain material groups as those most likely to be unaffected by the exposures and certain groups that will show changes in the properties, even though the effects may not be deleterious. For instance, dacron materials showed an average of 6.3% reduction in tensile properties after heat compatibility, but not after vacuum exposure which makes it acceptable for the specific application. Viton "A" MIL-R-25897 group exhibited a 35% compression set after one month thermal vacuum exposure, making it unacceptable for its specific application. In general, this qualification program has provided the necessary data to give assurance that nonmetallic materials, having passed a thermal screening, will undergo little significant changes of physical properties after long term vacuum exposure at moderate temperatures.

REFERENCES

1. Journal of Applied Polymer Science, Vol. 16, pp. 2503-2510, 1972.

2. Central Research Laboratory, Red Wing, Minnesota, Patent No. 3,507,163, dated 21 April 1970.

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